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# PATENT SPECIFICATION

DRAWINGS ATTACHED

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COMPLETE SPECIFICATION

## Improvements in and relating to the production of Thin Layers on Bases

We, BALZERS PATENT- UND BETEILIGUNGS-AKTIENGESELLSCHAFT, a body corporate organised under the laws of the Principality of Liechtenstein, of 9496 Balzers, Principality of Liechtenstein, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to a method for the production of thin layers on bases, said bases being heated and, after having reached a predetermined temperature, being deposited upon with the substance of the layer *in vacuo*. It is known, that layers deposited on a heated base have in general a better adherence than those deposited on cold bases. Heating may be effected by electric glow discharge at sub-atmospheric pressure or by radiation heating by a heating element. What temperature the bases, which are in general of a glass-like nature, assume in this process, depends apart from the heat output also on the capacity for the absorption and emission of light and heat radiation, as well as on the conduction of heat away from the base through the holder device. The temperature ought to be adjustable as accurately as possible before the vapour deposition. Too low a temperature reduces the adherence of the layers subsequently to be deposited. On the other hand the bases to be deposited upon from the vapour phase, which often are sensitive optical components, cannot be heated at will without limitation.

In order to measure the temperature before the vapour deposition, mercury thermometers, bolometers and theroelectric couples have been used. All these thermometers have the disadvantage, but it is difficult to bring them into thermally conductive contact with the bases to be deposited upon from the vapour phase. When, however, heating is not effected by thermally conductive contact but by the absorption of radiation, it depends on the varying thermal emission of the bases to be deposited upon from the vapour phase, and thus becomes indeterminate.

The invention has the object of providing a method for surveying the temperature of the bases to be deposited upon from the vapour phase during the deposition of thin layers, which method allows a reliable determination of the moment, which is correct for beginning the vapour deposition after a preceding heating of the base. Thus faulty layers can be obviated and great savings can be made in the series production of thin layers.

The method according to the invention is characterised in that the attaining of the predetermined temperature and accordingly the earliest moment for beginning the vapour deposition is ascertained by measuring continuously the electrical insulation resistance of a base not yet deposited upon while being heated.

Although it has been known to measure continuously the electric resistance of a thin metal layer growing on a base during the vapour deposition and to interrupt the vapour deposition, when a certain resistance value of the layer is reached, which corresponds to a certain thickness of the layer, the continuous measuring of the electrical resistance did not serve in this case for adjusting the temperature, and was carried out on the layer itself and not on the base thereof. It had not been realised, that determining the resistance of the base for the layer, which in the cold condition constitutes an electric insulator of high insulation resistance of the order of magnitude of hundreds of meghoms, constitutes an excellently suitable means for continuously measuring the true temperature of the bases, which usually are partly permeable to light and heat radiation.

The method of continuous measurement proposed according to the invention is very sensitive, since the materials used for bases are in general ion conductors, having a temperature coefficient of electrical resistance, which is high in the temperature range of about

200°—400°C most important for the vapour deposition of thin layers, as it is shown in the subsequent table of the values of specific resistance of boron silicate glass (BK) and of silicate flint glass (SF) at various temperatures:—

| Temperature:— | 100 | 150 | 200 | 250 | 400 | °C. |
|---|---|---|---|---|---|---|
| BK—Glass | $3\times10^{13}$ | $3\times10^{12}$ | $2\times10^{11}$ | $8\times10^{4}$ | $3\times10^{3}$ | Ohm.cm |
| SF—Glass | $4\times10^{14}$ | $3\times10^{13}$ | $3\times10^{12}$ | $4\times10^{6}$ | $1\times10^{5}$ | Ohm.cm |

In the series vapour deposition of thin layers on optical components it has been found convenient to carry out the continuous measuring, instead of on these components themselves, on a so-called test-glass, which is arranged in a manner known *per se* in a similar manner as the components to be deposited upon, consists of the same material as these components, and is exposed to heating before the vapour deposition in the same manner.

In the accompanying drawing, Fig. 1 is a perspective view of a test-glass plate,

Fig. 2 is a graph plotting electic resistance in Ohm against temperature on the base in °C.

For example, as shown in Fig. 1, two opposite sides of a square test-glass plate 1 may be provided with a strip 2, 3, respectively, of aluminium or gold deposited thereon from the vapour phase. These strips, serving as electrodes, are connected to a resistance-measuring and -indicating device arranged outside the deposition plant. Since glass is an ion conductor those resistance measuring instruments are preferred in which the direction of the current measured is reversed time and again, in order to obviate a disturbing polarisation.

Fig. 2 shows by way of example the dependence of the electric resistance between two aluminium electrodes, 25 mm long and 10 mm wide each, which had been applied at a distance of 30 mm on a test-glass plate of 1 mm thickness and 25 × 50 mm in size, while no layers were deposited on the glass from the vapour phase. When vapour-depositing a layer, the resistance may change discontinuously when the layer material has a higher electric conductivity than the base. Measuring the electric resistance of layers deposited from the vapour phase is outside the scope of the present invention.

It will be seen in Figs. 2, that the resistance drops from about $8 \times 10^{10}$ at 100°C to about $6 \times 10^{5}$ at 400°C.

It has been found with electrodes deposited from the vapour phase over an area and connected to the base by molecular binding forces that better reproducible values of the insulation resistance are obtained than when the bases are merely clamped between the measuring electrodes. The arrangement of the electrodes on the base is not critical *per se;* it is only necessary to ascertain for a certain arrangement on a certain base and for the production of a certain layer once and for all by a preliminary test, which value of resistance indicates the temperature desirable for the vapour deposition intended. The invention can be readily applied to automatic vapour deposition plants, wherein control devices may be used, which permit vapour deposition only, when the resistance value of the base surveyed has dropped to a predetermined value.

WHAT WE CLAIM IS:—

1. A method for the production of thin layers on bases, wherein said bases are heated to a predetermined temperature and the layer substance is deposited thereon, after said temperature is reached, by vapour deposition *in vacuo,* the reaching of the predetermined temperature and accordingly of the earliest moment suitable for beginning the vapour deposition being ascertained by continuously measuring the electric insulation resistance of a base, on which no layer is deposited yet, while said base is being heated.

2. A method according to claim 1, wherein the insulation resistance of a test-glass is measured between two electrodes deposited thereon from the vapour phase.

3. A method according to claim 1, wherein the direction of the measuring current is alternately reversed while measuring the insulation resistance of the base.

4. A method according to claim 1, substantially as herein described.

5. Thin layers on bases when produced by a method according to any of the preceding claims.

6. A test-glass substantially as herein described, when used in a method according to any of the claims 1—4.

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Fig. 1

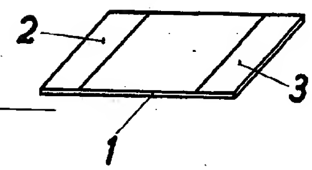


Fig. 2